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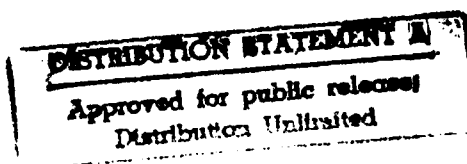
**TECHNICAL CONTRIBUTORS:** Dr. Aly Fathy, David Kalokitis,  
Valerie Pendrick,  
Dr. K. S. Harshavardhan (Neocera),  
Dr. Albert Piqué (Neocera),  
Dr. Erwin Belohoubek,  
Dr. T. Venkatesan (Neocera)

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### Summary

YBCO on  $\text{MgF}_2$  withstood post annealing to  $750^\circ\text{C}$  without deterioration. This allows the deposition of high quality multiple layer YBCO films onto both sides of a  $\text{MgF}_2$  substrate.

$\text{GdBaCuO}$  films were deposited onto  $\text{LaAlO}_3$  and appear to be superior to YBCO in terms of lower particulate density, slightly higher  $T_c$ 's, and higher critical current density. The ramifications could be very beneficial to the MCM program. Furthermore, the tolerance of these films to a wider range of deposition conditions indicates a possibility of using these films on  $\text{MgF}_2$ .

A new processing method is proposed for making a thin low dielectric constant HTS microstrip structure with a glass/metal handle that is suitable for both MCM and millimeter wave applications. The main advantage of this procedure is that the HTS films are deposited onto a high quality, single crystalline bulk substrate that is mechanically and chemically thinned. No thick epitaxial  $\text{MgF}_2$  layer has to be grown in conjunction with other multi-layer YBCO/dielectric layers. This process is considered an interim approach for MCM HTS circuits until high quality, stacked, multilayer depositions of HTS films and dielectrics can be routinely achieved.

A process for achieving robust thick gold contacts to YBCO was successfully demonstrated. Fine line definition of YBCO patterns (down to  $5\mu$ ) on  $\text{MgF}_2$  was achieved using an ion milling technique. Non-superconducting metal in the form of Ti-Au was successfully deposited onto  $\text{MgF}_2$  with good adherence, which allows wires or ribbons to be welded to the patterned lines. This allows comparisons to be made between YBCO and normal metal sample circuits.

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## I. HTSC MATERIAL DEVELOPMENT

### A. Post Annealing of YBCO Films Deposited on $\text{MgF}_2$ Substrates

Post annealing experiments were carried out to determine whether YBCO films on  $\text{MgF}_2$  heated to  $700^\circ\text{C}$  and above would survive without any deterioration. This is important for multi-layer YBCO films that must be deposited on opposite sides of the substrates. Degradation at relatively high temperatures is possible due to the highly reactive nature of the  $\text{MgF}_2$  substrate at these temperatures.

Fig. 1 summarizes the results with these post annealing experiments. The figure presents x-ray diffraction data on three different films annealed for 30 minutes at 800 C, 750 C, and 700 C, respectively. It may be seen from these results that films annealed at 800 C undergo severe degradation in their structural properties as revealed by additional peaks which do not conform to the standard (001) peaks. The (001) peaks are rather weak, which indicates significant structural deterioration of the film. The films have  $T_c$ 's of  $\sim 88$  K before annealing. After annealing at 800 C, the films do not show any  $T_c$  above 77 K. Films annealed at temperatures of 750 and 700 C, on the other hand, do not show any detectable chemical reaction with the substrate as revealed by the presence of predominately (001) ( $l=1,2,3,\dots$ ) reflections in their x-ray profiles. Further, the films retained their superconducting properties (as measured by ac susceptibility, Fig. 2), without any significant deterioration in their  $T_c$ 's. Annealing at 800 C deteriorates film surface whereas annealing at 750 C and 700 C did not show any degradation as revealed by their corresponding optical micrographs (Fig. 3).

These results suggest that *ex situ* post annealing of YBCO films at temperatures as high as  $750^\circ\text{C}$  will not deteriorate the surface and superconducting properties of the YBCO films. Therefore, it should be possible to achieve high quality YBCO films on both sides of the  $\text{MgF}_2$  substrate.

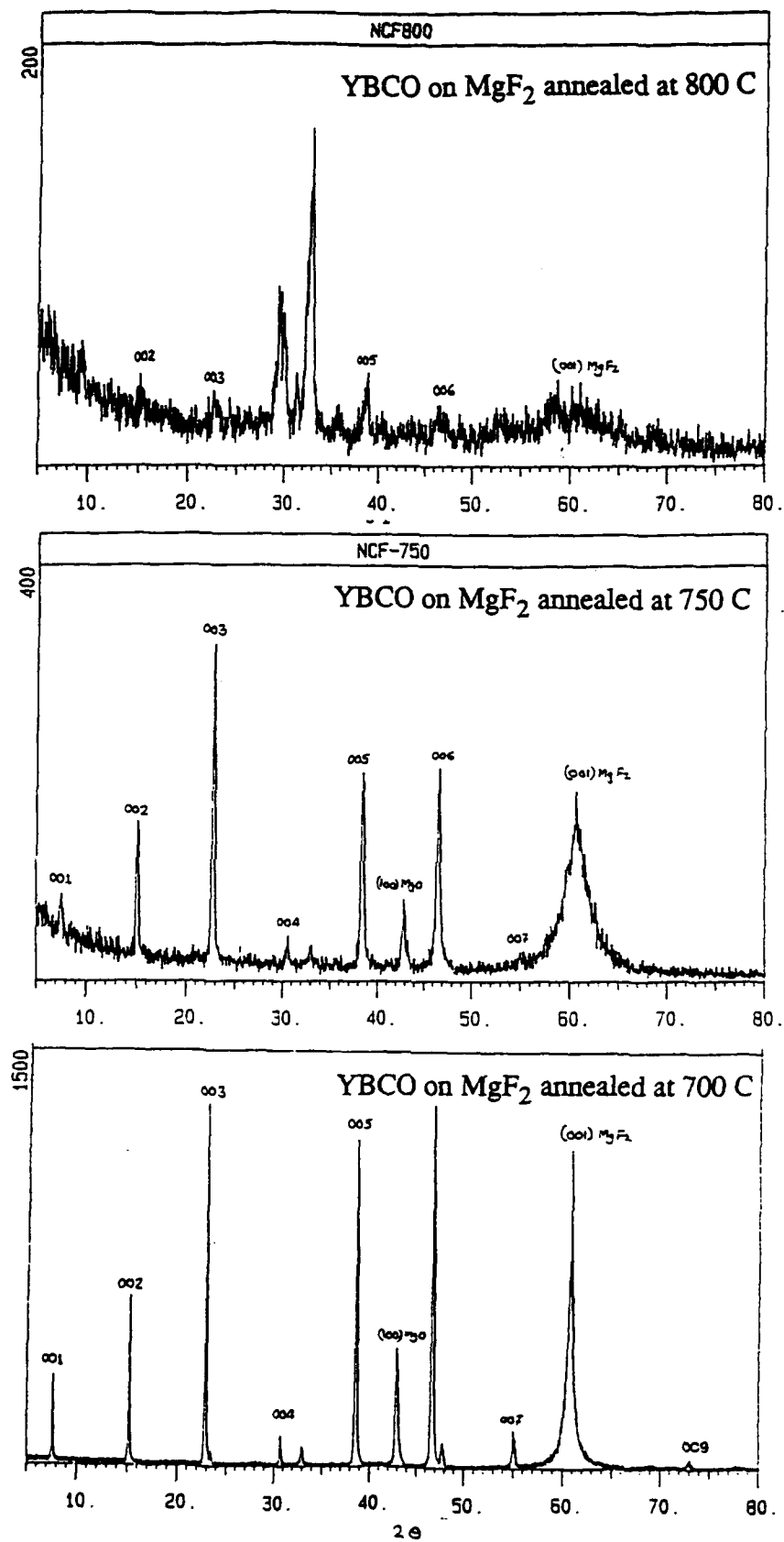


Fig. 1. Post Annealing Experiments with YBCO on MgF<sub>2</sub> Substrates.

## YBCO post annealed at 750 C

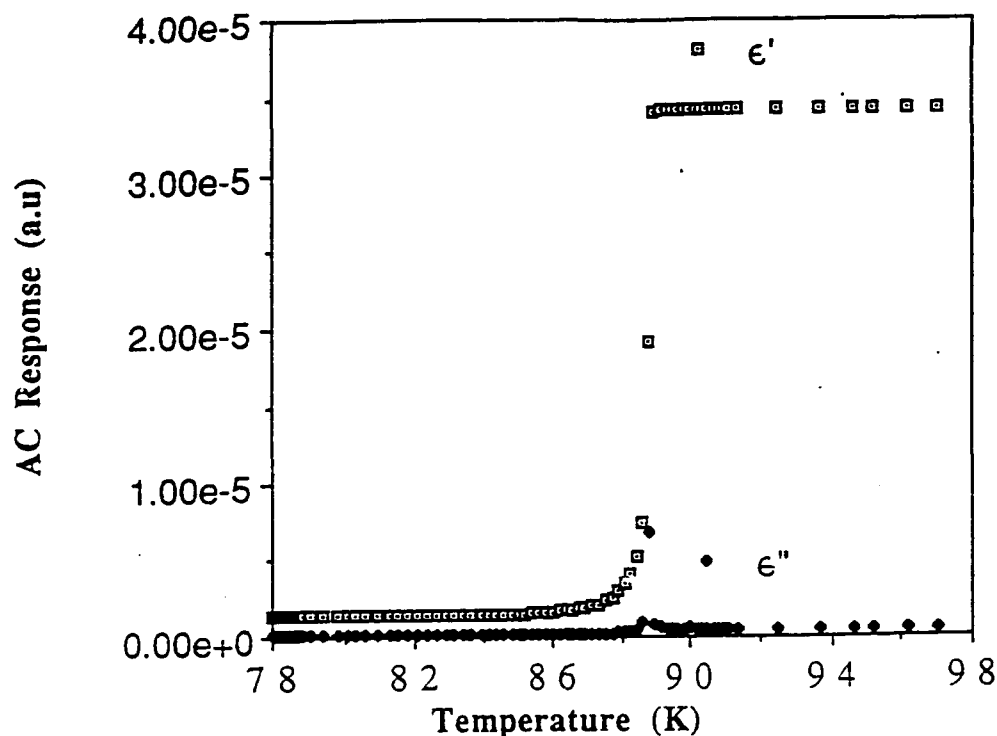
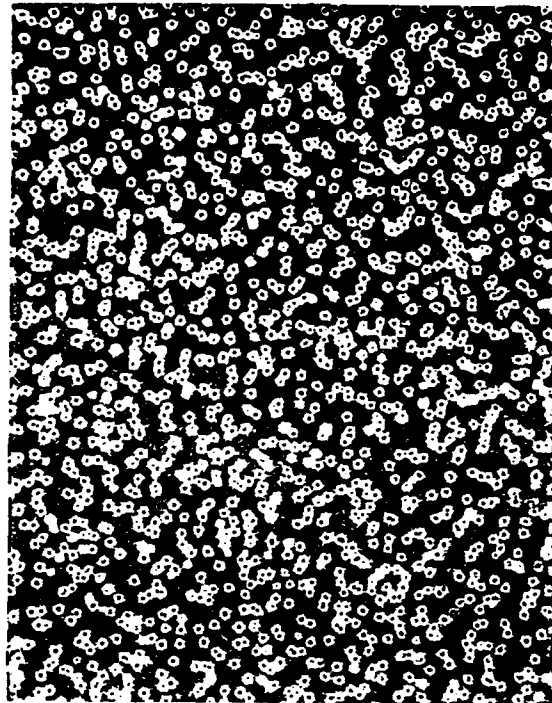


Fig. 2. AC Susceptibility of YBCO Post Annealed at 750C.

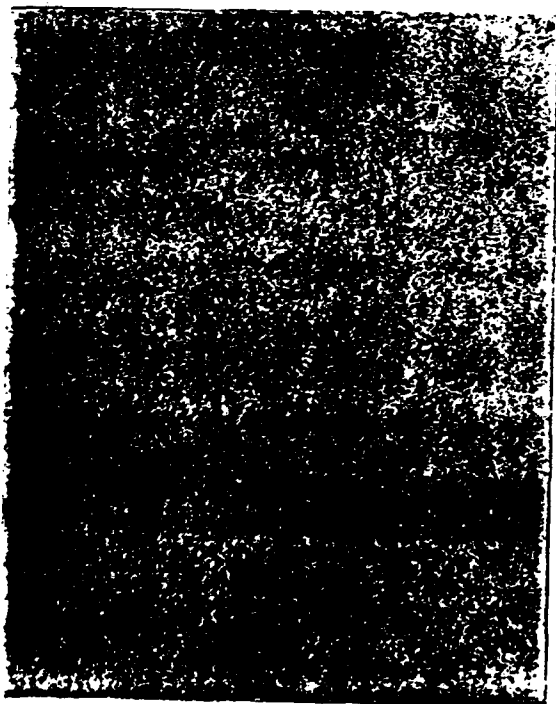
### B. Exploration of GdBaCuO as a Possible Alternative to YBCO

Until now only YBCO films have been deposited epitaxially on the  $\text{MgF}_2$  substrates. To grow high quality films reproducibly on  $\text{MgF}_2$  substrates, we found that a very strict control of the deposition parameters during the film growth is critical. It may be mentioned here that YBCO films on  $\text{MgF}_2$  substrates are grown at relatively lower temperature (75-100 C lower) compared to temperatures at which films are grown on  $\text{LaAlO}_3$  and sapphire. These lower temperatures are mandatory due to the extreme chemical reactivity of the substrate at higher temperatures. It is recently reported<sup>1,2</sup> that the GdBaCuO system is more tolerant to the processing conditions both in the bulk and thin film forms. Also, the  $\text{GdBa}_2\text{Cu}_3\text{O}_7$  system exhibits the highest  $T_c$  of 94 K among all other rare earth (Re) substitutions of the  $\text{ReBa}_2\text{Cu}_3\text{O}_7$  family.

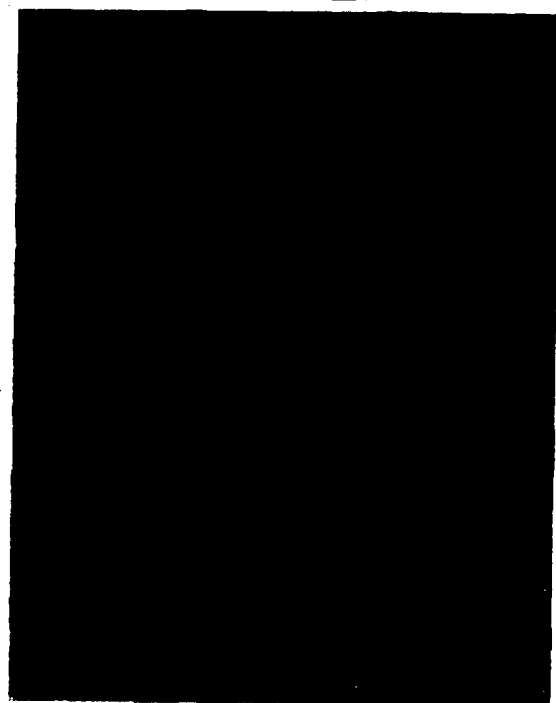
Our preliminary depositions carried out on  $\text{LaAlO}_3$  substrates yielded  $T_c$ 's of ~ 91-92 K. One interesting observation is that GdBaCuO films contained a much lower density of particulates and had smoother surfaces



YBCO on  $\text{MgF}_2$  annealed at 800 C ( $\times 500$ )



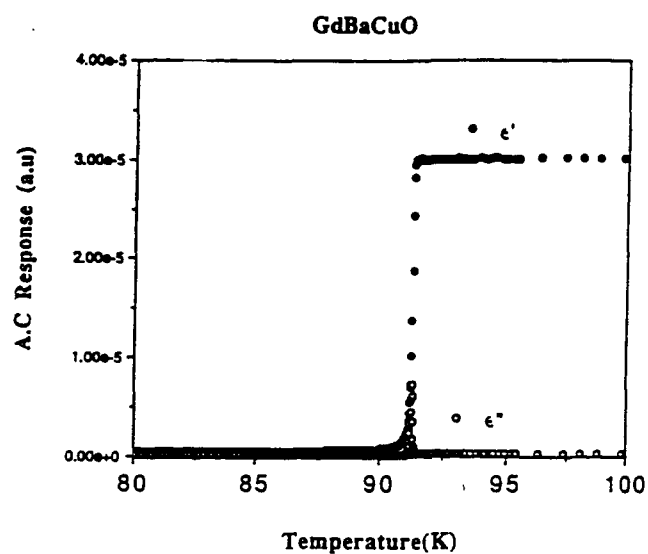
YBCO on  $\text{MgF}_2$  annealed at 750 C ( $\times 500$ )



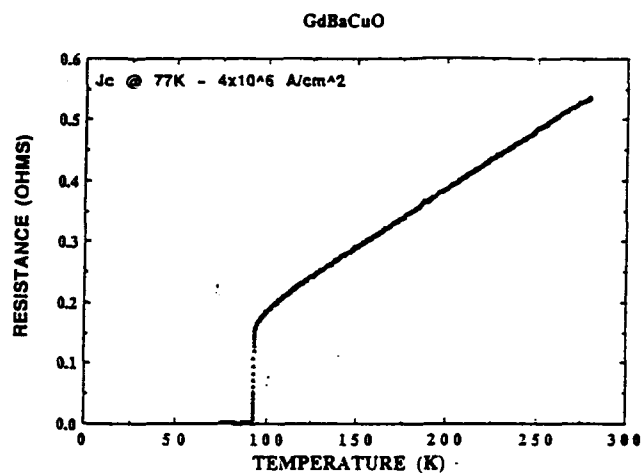
YBCO on  $\text{MgF}_2$  annealed at 700 C ( $\times 500$ )

**Fig. 3. Film Surface of YBCO on  $\text{MgF}_2$  post annealed at 700C, 750C, and 800C.**

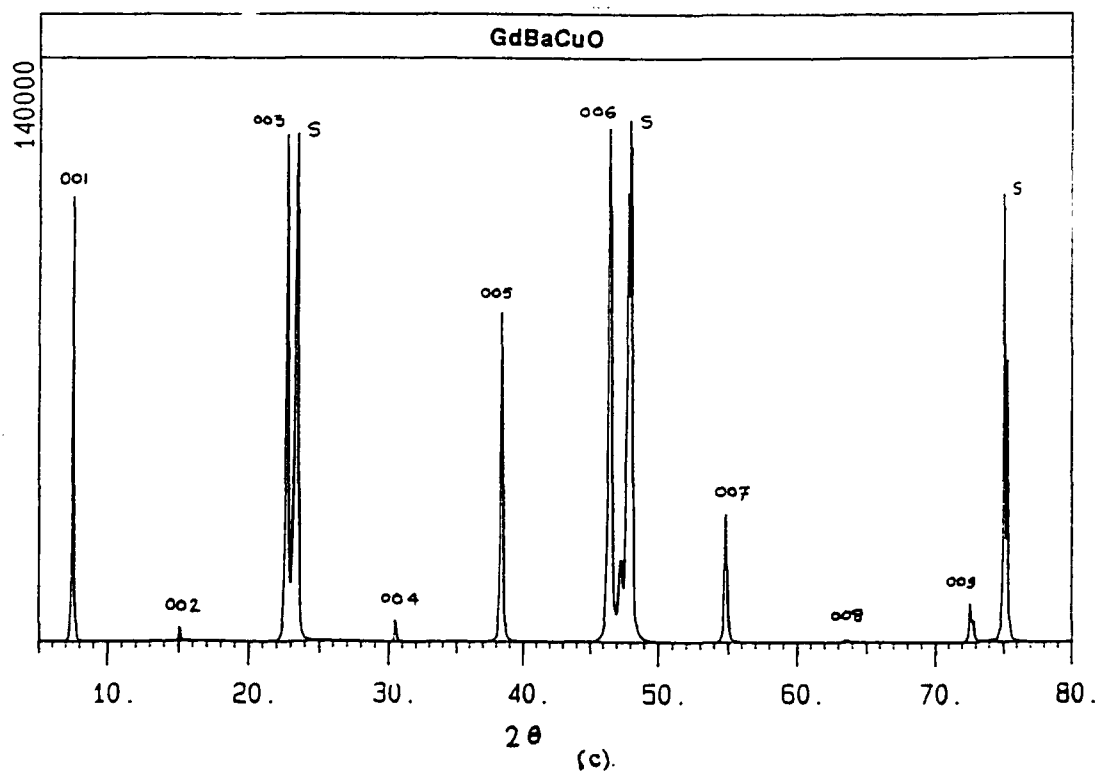
than YBCO films. The growth kinetics of GdBaCuO and YBCO films appear to be different and needs to be explored at a later stage. The results obtained on GdBaCuO films are compiled in Fig. 4. Figs. 4a and 4b show the  $T_c$  of the film measured by ac susceptibility and resistivity measurements, respectively. This data indicates the quality of the films is very high. Critical current density of the films as measured on 70 $\mu$ m wide bridges are in the range of  $4 \times 10^6$  A/cm<sup>2</sup> at 77 K. The films are c-axis oriented as revealed by their x-ray diffraction profiles (Fig. 4c). The microwave properties are being studied. The high critical current densities, higher  $T_c$ 's, and superior surface features (smoothness) and tolerance of these films to deposition conditions indicate a possibility of using these films in conjunction with the MgF<sub>2</sub> substrates as well. If GdBaCuO films are proven to be superior to YBCO films, the ramifications could be very beneficial for the upcoming MCM program, particularly in achieving a low particulate density (smooth surfaces) and high critical current densities at 77 K. The initial experimental results we presented here indeed point out to such a possibility.



(a)



(b)



**Fig. 4. Measurement results with GdBaCuO films grown on  $\text{LaAlO}_3$ . (a) AC Susceptibility; (b) Resistance; and (c) x-ray Diffraction Profiles.**

## **II. HTS Circuit Fabrication**

### **A. Proposed Circuit Structure for MCM/Millimeter Wave Applications**

For both millimeter wave circuit and multi-chip module (MCM) applications, it is desirable to have microstrip transmission line structures that are thin and use a low dielectric constant substrate. Magnesium fluoride has a very attractive low dielectric constant of about 5. The challenge is to develop a good means of achieving a substrate thickness of only a few microns with high quality superconductors on both sides. Fig. 5 shows the structure that is needed for an MCM module using superconductors. A straight-forward approach for fabricating such a structure would be to sequentially deposit the seven single crystalline layers of superconductors and dielectrics on top of a thick host substrate such as YSZ. This is extremely difficult to achieve without degradation of the quality of the superconducting films, especially the most important signal lines on top of the structure that are only 2 microns wide and require a high critical current density. In addition, good heat conductivity from the superconducting film layers to the metal base of the MCM hermetically sealed module is needed to minimize the required  $T_c$  of the HTS films. A proposed processing method for achieving the desired structure with the added benefit of the support (handle) substrate being largely metal for maximizing heat conductivity is shown in Fig. 6. The principal features are the attachment of a glass/metal handle to a YBCO covered magnesium fluoride substrate and a subsequent thinning of the latter to the desired thickness of a few microns by a combination of mechanical and chemical means. The critical signal conductor lines are the last to be deposited onto the thinned bulk magnesium fluoride substrate. The main advantage of this procedure is that the superconducting films are deposited onto a high quality, single crystalline substrate. No thick epitaxial magnesium fluoride layer has to be grown as with the original straightforward approach using stacked multilayer depositions.

In order to find the acceptable magnesium fluoride thickness for the MCM application, the electrical parameters of signal line characteristic impedance and cross coupling between adjacent signal lines must be considered. Fig. 7 shows the relationship between characteristic impedance and the microstrip width ( $w$ ) and substrate thickness ( $h$ ). Since the device impedance in an MCM module is nominally 100 ohms and the

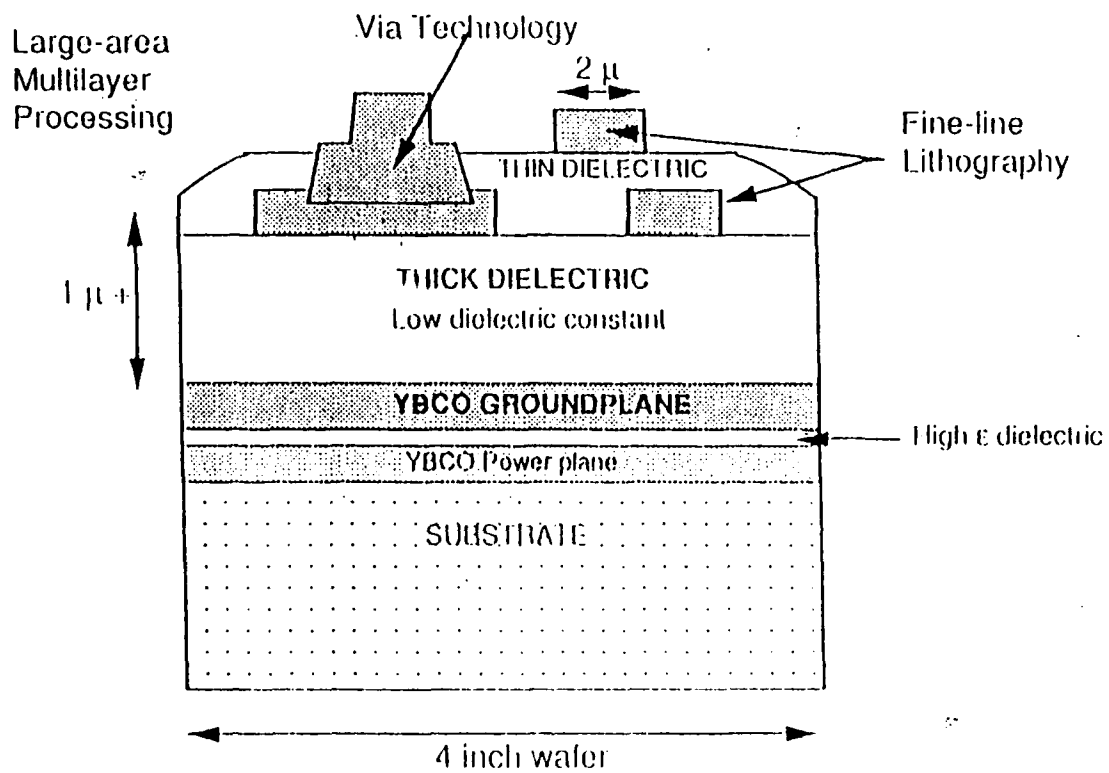
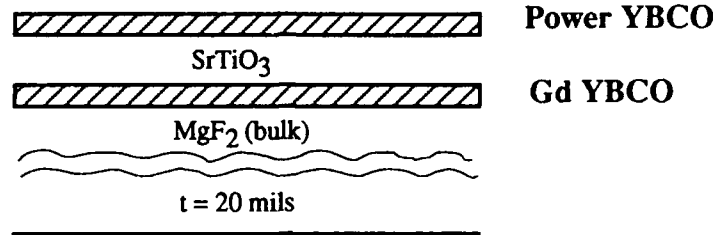
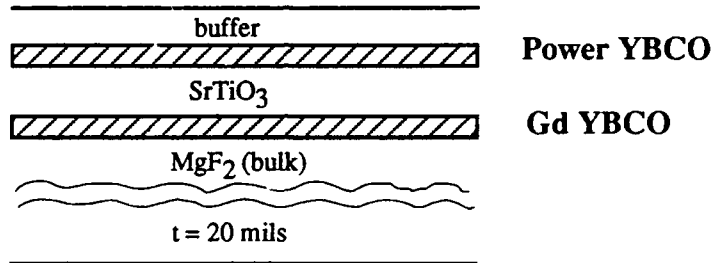


Fig. 5. MCM Structure

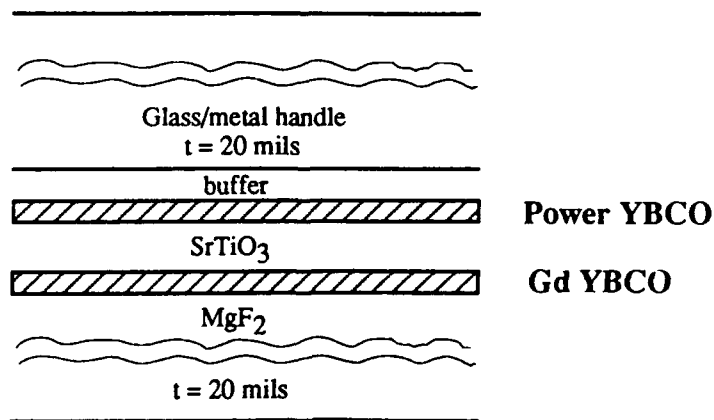
**Step 1** Grow ground and power plane YBCO layers onto bulk  $\text{MgF}_2$  substrate.



**Step 2** Deposit buffer onto power YBCO surface.

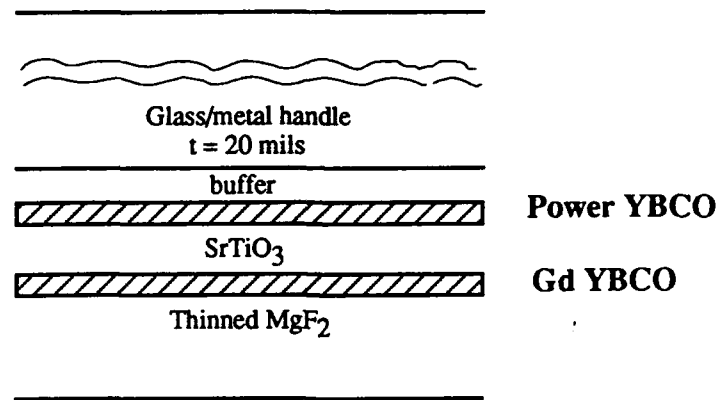


**Step 3** Attach thick glass/metal handle to buffer surface.

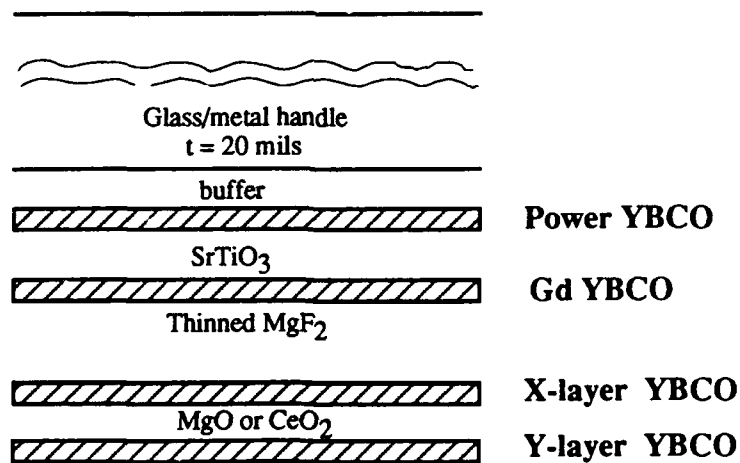


**Fig. 6. Proposed Fabrication Process**

**Step 4** Thin  $\text{MgF}_2$  to 10 microns using mechanical and/or chemical etching method.



**Step 5** Grow X and Y YBCO layers onto  $\text{MgF}_2$  substrate.



**Fig. 6. Proposed Fabrication Process (Con't)**

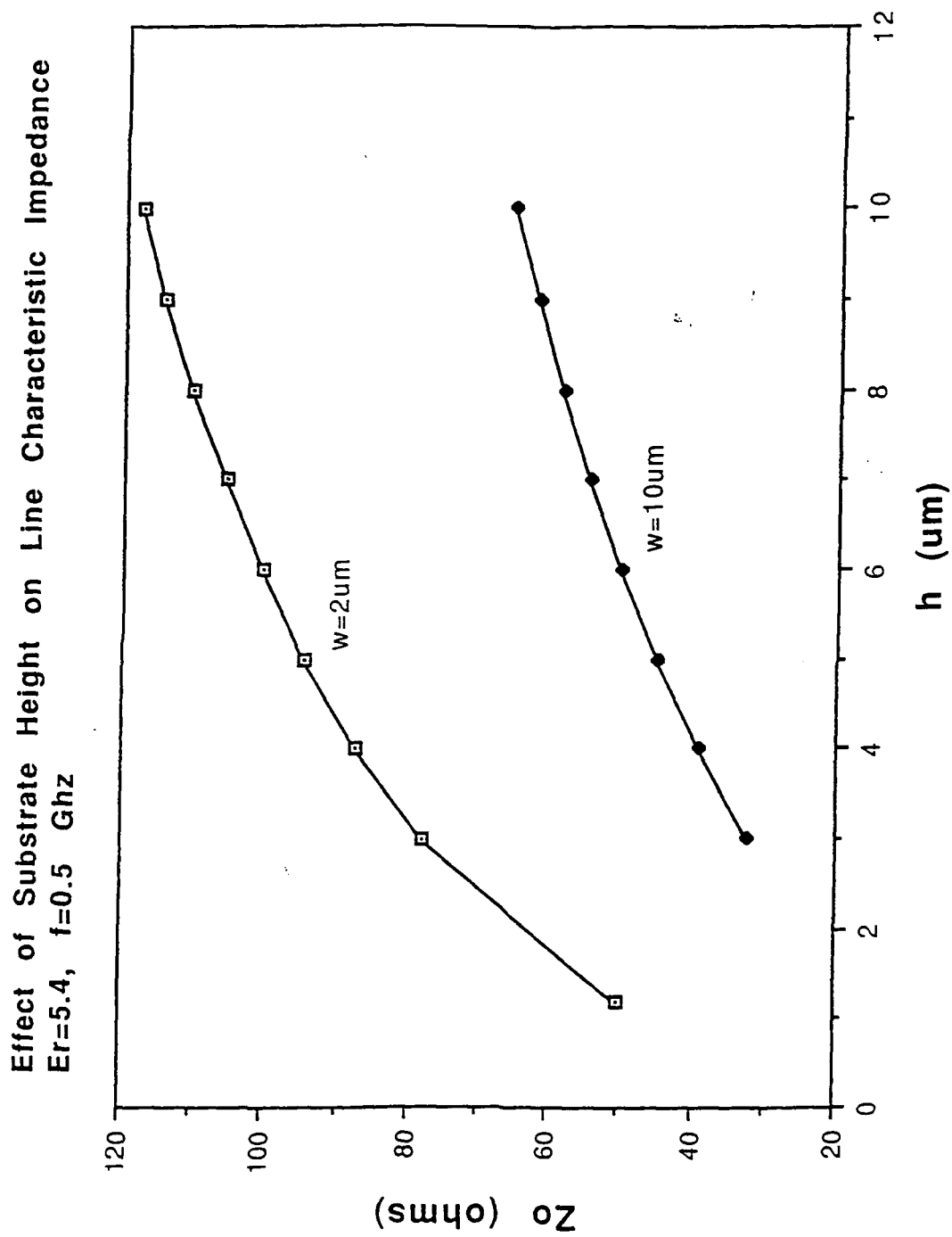


Fig. 7. Microstrip Line Characteristic Impedance vs. Substrate Thickness ( $h$ ) for Various Line Widths ( $W$ ).

sensitivity of the impedance to  $h$  becomes increasingly less at high impedance levels, an impedance level above 80 ohms and a substrate thickness greater than 3 microns is desirable. Assuming an allowed cross coupling of 25 dB between lines spaced 8-12 microns apart, the curve of coupling versus line spacing ( $s$ ) normalized to the substrate thickness (see Fig. 8) indicates a maximum allowed substrate thickness of about 6 microns.

For a millimeter wave circuit such as a delay line, the desirable characteristic impedance is 50-70 ohms and the line width can be wider than for the MCM case. Assuming a line width of 10 microns with an allowed 25 dB cross coupling between adjacent lines spaced 20-30 microns center to center, the substrate thickness should be 6-10 microns thick.

For application to both MCM and mm-wave circuits, a reasonable goal for thinning the magnesium substrate is chosen to be 6 microns. A proposed structure for demonstrating the process is shown in Fig. 9. It replaces the two x-y signal lines for the MCM application by one signal line and eliminates the power plane line. With this structure we can make a demonstration circuit in the form of a meander line resonator that can be tested from 1 GHz to 26 GHz for both loss as well as its performance as a delay line. A meeting at ARPA has been scheduled for May 5, 1993, to discuss the proposed approach and emphasize its importance to both the current MCM program and to future millimeter wave applications.

## **B. Glass/Metal Handle**

Before attaching the glass/metal handle in the processing procedure shown in Fig. 6, a buffer layer must be grown onto the YBCO-covered magnesium fluoride sample to serve as protection from chemical interaction between the glass/metal and the YBCO film. A first choice for such a buffer is MgO because of previous Neocera experience. The glass/metal handle will be composed of a thick metal base of copper/#409 stainless steel/copper covered with a thin glass bonding layer of crystalline lead zinc borate. Both materials have a thermal expansion coefficient of about  $10 \times 10^{-6}$ , which closely matches that of the YBCO and the magnesium fluoride. The glass/metal handle is attached to the buffer layer surface of the YBCO-magnesium fluoride sample at about 500 degrees C and because of its crystalline nature will withstand subsequent heating of 725 degrees C needed for the final top YBCO signal layers.

### Coupling versus Spacing of Signal Lines

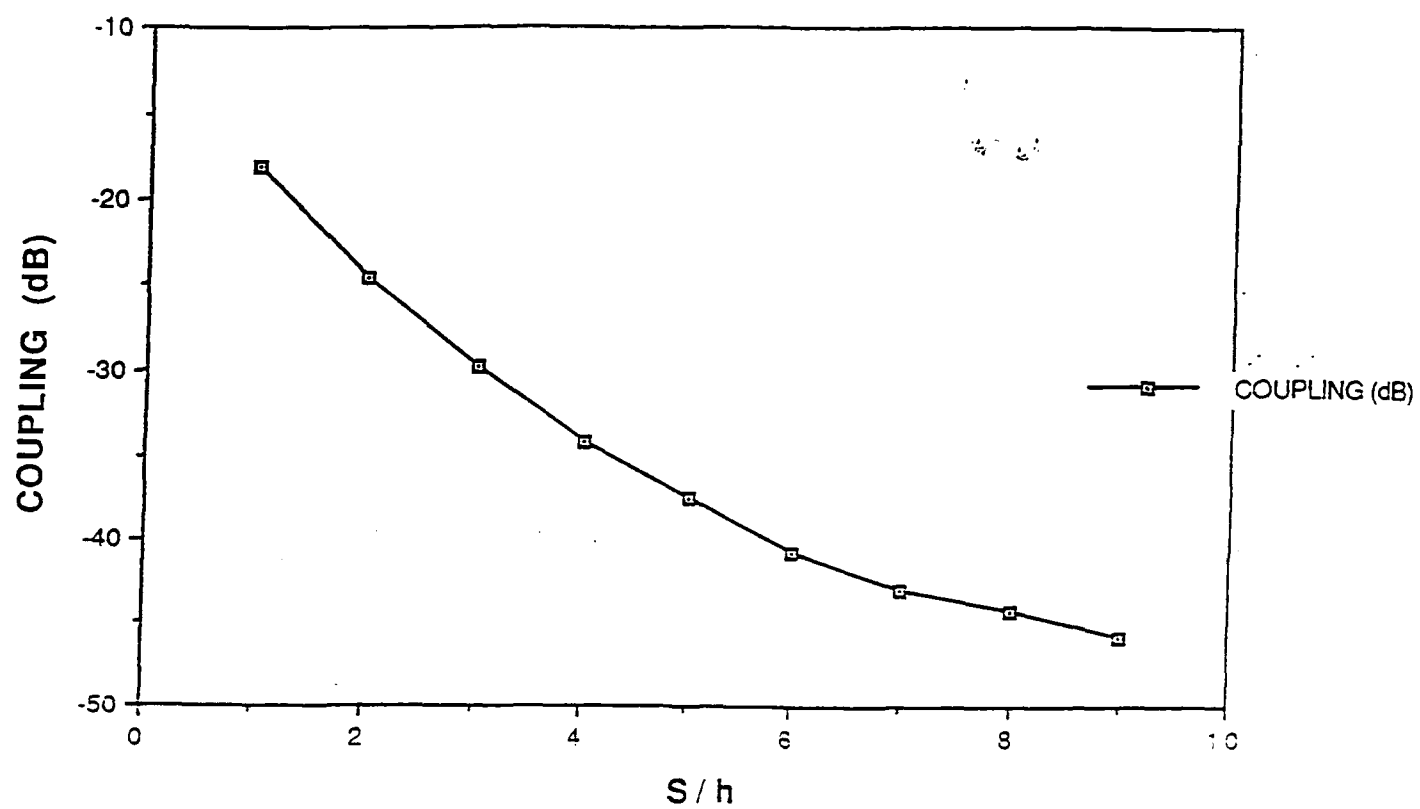


Fig. 8. Coupling between Microstrip Lines vs. Spacing (S) Normalized to Substrate Height (h).

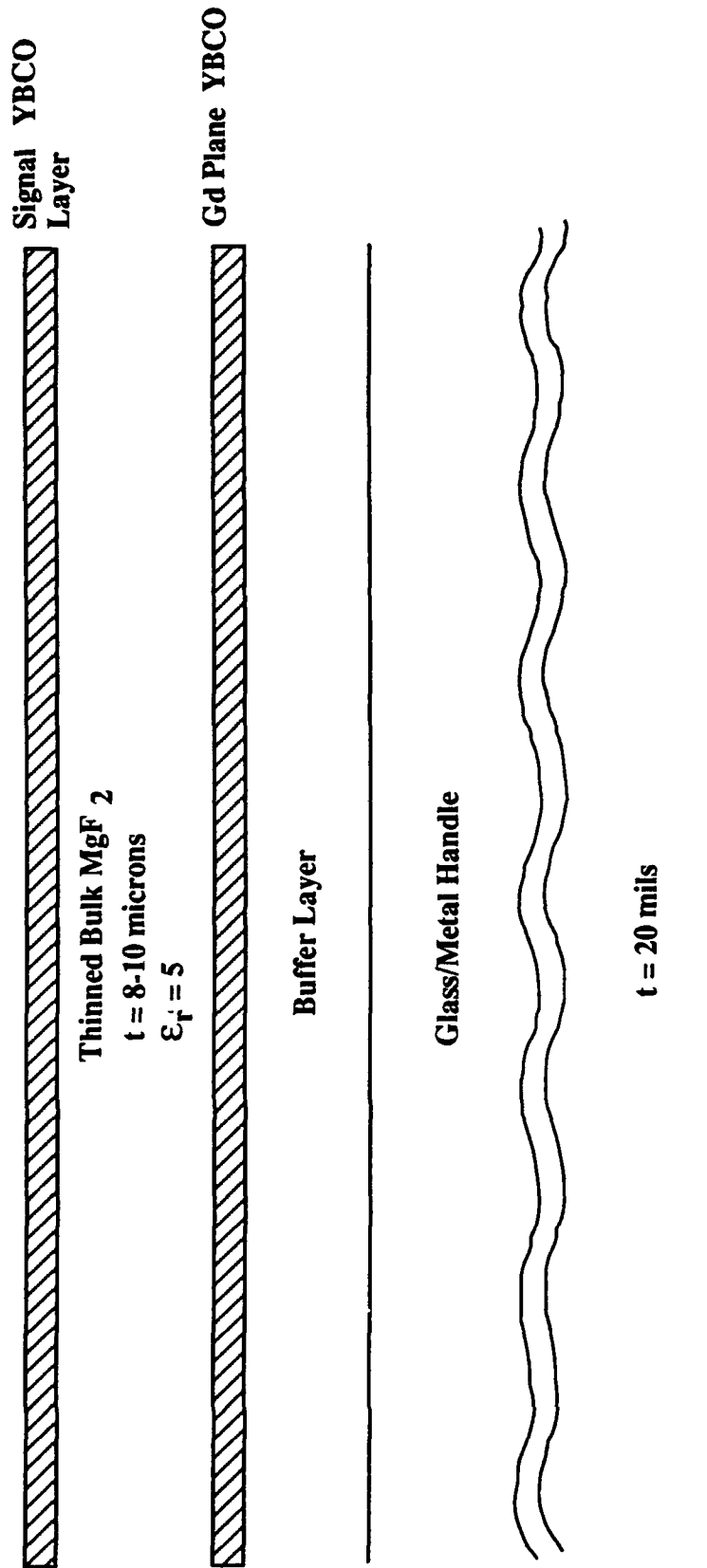


Fig. 9. Proposed Demonstration Structure for MCM/Millimeter-Wave Application

### **C. MgF<sub>2</sub> Polishing**

During this past quarter we investigated the thinning of single crystal MgF<sub>2</sub> wafers from 0.254mm to less than 25μm through mechanical lapping and chemical polishing. The wafers are purchased from the manufacturer in 2.54 x 2.54cm squares and are approximately 0.254mm thick. The wafers were lapped first on a tin lap embedded with diamond to remove as much of the material as possible. This resulted in a thickness reduction from 254μm to 13.5μm. Although we could significantly reduce the thickness with this method, lapping does not leave an epitaxial finish on the sample. Next, the wafers were chemically polished with Rodel semiconductor polishing slurry to remove all scratches and further reduce the thickness. The resulting finish on the samples is better than that provided by the manufacturer. The final thickness of the samples was 10μm. The sample thickness are checked by a Tencor brand profilometer.

### **D. Fine Line Definition**

Test circuits with line widths less than 10μm were successfully patterned using ion milling in Ar<sup>+</sup>. Samples were 1 cm x 1 cm MgF<sub>2</sub> and coated on one side with SrTiO<sub>3</sub> and MgO buffer layers as well as 3000Å of YBCO. A test mask with 5, 6, and 7 μm was used. Two samples were successfully patterned using ion milling.

### **E. Non-superconducting Test Circuits**

Work continued on nonsuperconducting circuit fabrication for use as a comparison with YBCO sample circuits. Test circuits were successfully fabricated using Ti-Au. This differs from our previous metallization layers which were Ti-Cu-Ni-Au. The copper layer created stress in the metal layers causing them to lift from the MgF<sub>2</sub>. Wires were soldered to the new circuits and ribbons were successfully welded. Further work in this area is being discontinued in favor of demonstrating the new glass/metal handle approach that is more applicable to MCM development.

### **F. Gold Contacts to YBCO**

Work continued on developing an economical yet robust method of contact fabrication. Two methods for fabricating gold contacts on YBCO were investigated. Using a standard lift-off technique, we were able to

fabricate contacts with 0.5 $\mu$ m of gold. A modified lift-off technique was employed to define thicker contacts of 1.3 $\mu$ m of gold. Although the 1.3 $\mu$ m of gold requires additional steps, the contacts should be more robust.

The standard lift-off method starts with definition of the YBCO by ion milling in Ar<sup>+</sup> as shown in Figs. 10 and 11. Photoresist is then applied to the sample, exposed and developed to form the contact pattern, shown in Fig. 12. Fig. 13 illustrates the sample with 0.5 $\mu$ m of sputter deposited gold. (The gold layer is kept relatively thin to allow for lift off.) The unwanted gold is removed by soaking the sample in acetone. The final circuit is shown in Fig. 14. The gold only adheres to the YBCO in the contact area; the photoresist prevents the gold from adhering in the other area. Contacts made with this technique were strong enough to withstand the bonding of gold ribbons. This method is relatively fast and simple.

For the modified lift-off method, the YBCO is also defined using ion milling and the contact area defined using photoresist as illustrated for the standard method (Fig. 10, 11, 12). However, a thicker gold layer (1.3 $\mu$ m instead of the previous 0.5 $\mu$ m) is sputter-deposited onto the sample structure shown in Fig. 13. Photoresist is applied to the sample again on top of the blanket-coated gold, exposed, and developed to cover only the contact area as shown in Fig. 15. This protects the contacts and leaves the rest of the gold exposed. The unwanted gold is removed by ion milling in Ar<sup>+</sup> as illustrated in Fig. 16. The first layer of photoresist protects the YBCO; the second layer protects the gold contacts. After ion milling, both layers of photoresist are removed in acetone to produce the final result shown in Fig. 14. This method produced contacts that were also strong enough for the bonding of gold ribbons. However, the additional gold should hold up better than the thin gold in the event of reworking the sample. This method does involve additional steps, but they are low risk.

## Gold Contact Fabrication by Lift Off Method

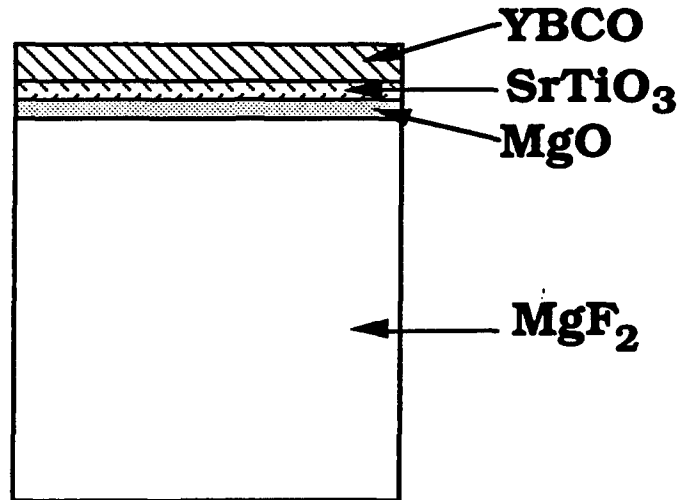


Fig. 10. Standard Sample.

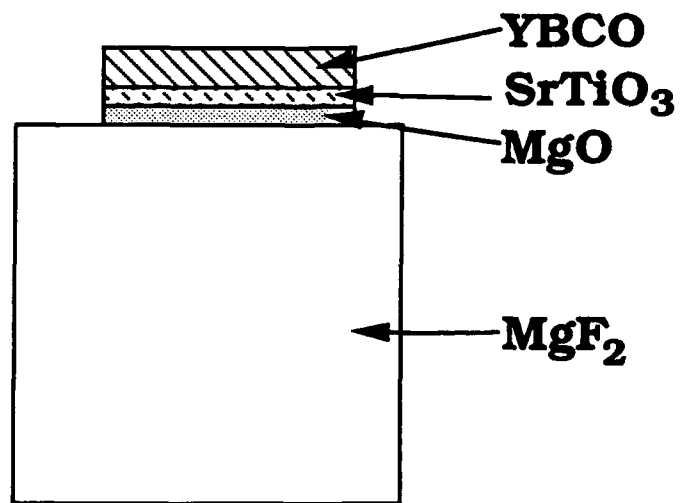


Fig. 11. YBCO and buffer layers defined using ion milling.

## Gold Contact Fabrication by Lift Off Method (cont.)

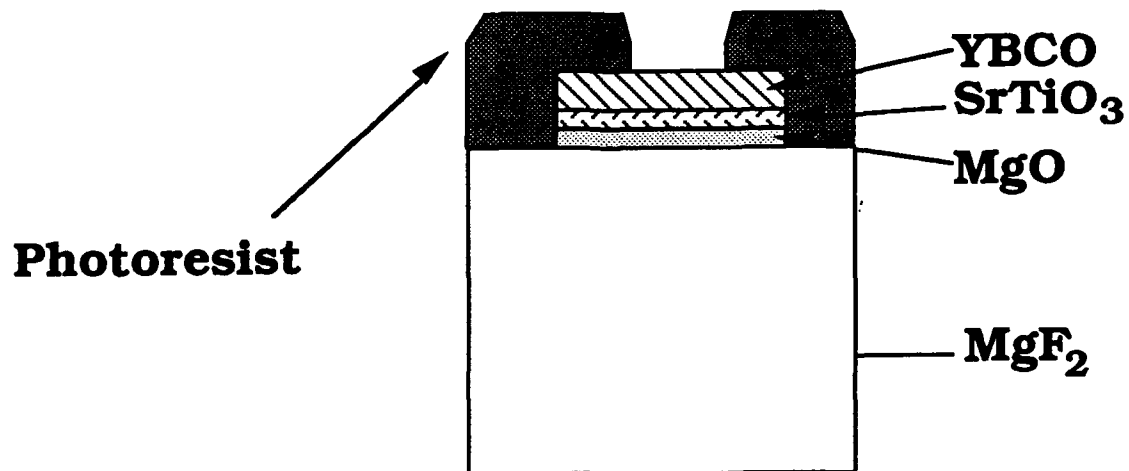


Fig. 12. Photoresist defined for contact pattern.

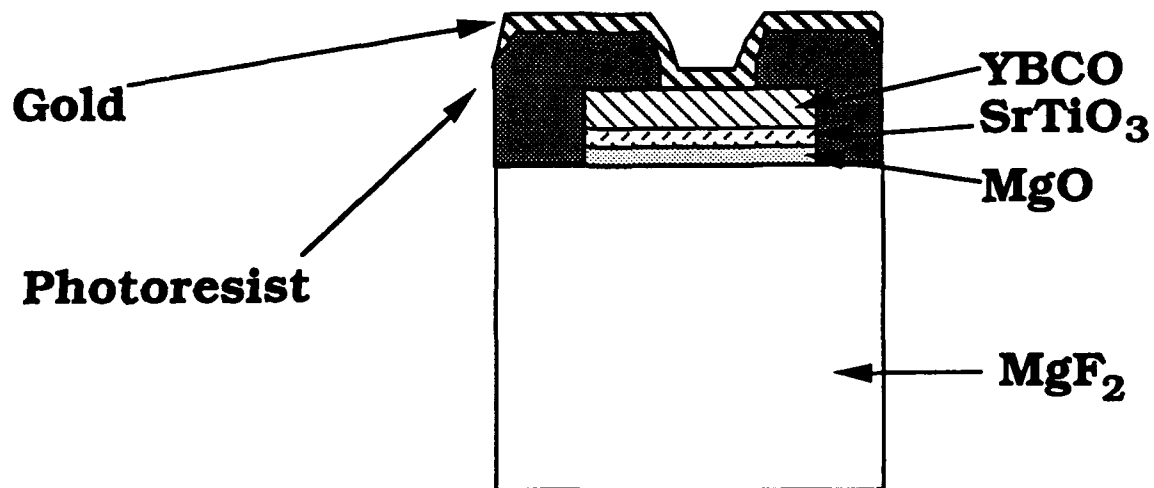
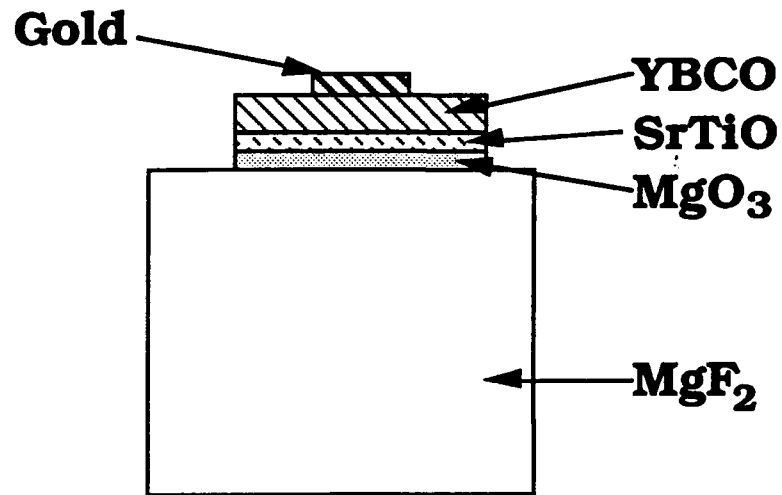


Fig. 13. Gold layer added for contacts.

## Gold Contact Fabrication by Lift Off Method (cont.)



**Fig. 14.** Unwanted gold 'lifted off' to form contacts.

## Gold Contact Fabrication by Modified Lift Off Method (cont.)

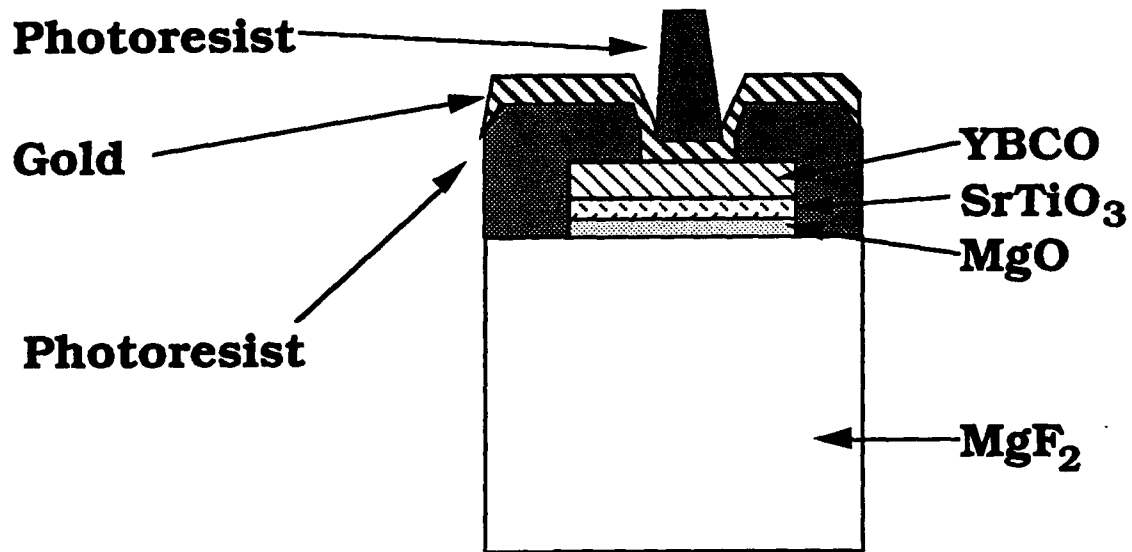


Fig. 15. 2nd photoresist layer defined to protect contact area.

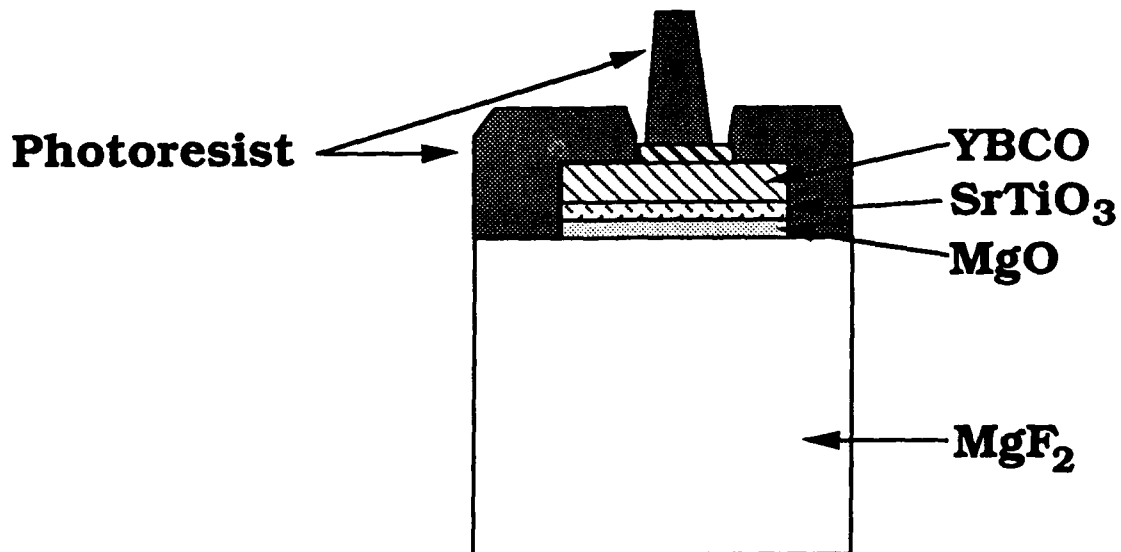


Fig. 16. Unwanted gold removed by ion milling.

**V. REFERENCE**

1. M.W. Shin et al. J. Mater Res. 7, 3194 (1992).

**VI. CHANGE IN KEY PERSONNEL: None****VII. SUMMARY OF SUBSTANTIVE INFORMATION DERIVED FROM SPECIAL EVENTS: None****VIII. PROBLEMS ENCOUNTERED AND/OR ANTICIPATED: None****IX. ACTION REQUIRED BY THE GOVERNMENT : None****X. FISCAL STATUS**

- |   |             |
|---|-------------|
| 1. Amount currently provided on contract: | \$900K      |
| 2. Expenditures and commitments to date:  | \$669K      |
| 3. Funds required to complete work:       | \$1,585,085 |